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(54)【発明の名称】　超微粉シリカ砥石およびその製造方法

(57)【要約】

【目的】　クラックの発生を極力防止でき、かつ砥粒の自生作用を活発に発現させつつ、加工生成物の逃げ場を増やし目づまりを未然に防ぐ、高い空孔率をもった超微粉シリカ砥石を提供する。

【構成】　フェノール樹脂によりコーティングされた超微粉シリカがプレス成形されて空孔率０～８０％に形成されたことを特徴としている。

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【特許請求の範囲】

【請求項１】　フェノール樹脂によりコーティングされた超微粉シリカがプレス成形されて空孔率０～８０％に形成されたことを特徴とする超微粉シリカ砥石。

【請求項２】　超微粉シリカに対するフェノール樹脂の割合が１～９９vol％であることを特徴とする請求項１記載の超微粉シリカ砥石。

【請求項３】　ビッカース硬さが１～４０Ｈｖであることを特徴とする請求項１または２記載の超微粉シリカ砥石。

【請求項４】　集中度が５０～３００であることを特徴とする請求項１、２または３記載の超微粉シリカ砥石。

【請求項５】　熱硬化性のフェノール樹脂を揮発性の溶媒に溶解した液中に超微粉シリカを加えてスラリー状にし、撹拌して均一に混合する工程と、該混合液体から揮発性成分を飛ばして乾燥させる工程と、乾燥物を粉砕して、超微粉シリカがフェノール樹脂によりコーティングされた粉末状の成形材料を形成する工程と、真空状にした容器内で前記成形材料を金型により所要形状にプレス成形する工程と、得られた成形体を焼成してフェノール樹脂を硬化させる工程とを具備することを特徴とする超微粉シリカ砥石の製造方法。

【請求項６】　金型から成形体を取り出す際に成形体に与圧をかけながら取り出すことを特徴とする請求項５記載の超微粉シリカ砥石の製造方法。

【請求項７】　前記成形材料をプレス成形する際の成形圧力が１０～６０００kgf/cm^2 であることを特徴とする請求項５または６記載の超微粉シリカ砥石の製造方法。

【請求項８】　超微粉シリカに対する熱硬化性のフェノール樹脂の割合が１～９９vol ％であることを特徴とする請求項５、６または７記載の超微粉シリカ砥石の製造方法。

【発明の詳細な説明】

【０００１】

【産業上の利用分野】本発明は超微粉シリカ砥石およびその製造方法に関する。

【０００２】

【背景技術】半導体ウェハーは高度の鏡面仕上げ精度が要求される。ナノメーターオーダーの仕上げ面粗さを得るためには、微細な砥粒からなる砥石を用いて研削を行うことが有効な手段である。これを実現するために微細砥粒をフェノール樹脂などの樹脂バインダーで固定した砥石の開発が行われているが、砥粒径が数～数１０ｎｍの超微粉（超微粒子粉末）を用いた砥石の製作はいまだ問題点が多く実現していない。

【０００３】超微粉の砥粒を用いた砥石が実現しない理由としては以下の原因があげられる。

①超微粒子粉末が有する特徴の１つである粒子の凝集・集塊のため、バインダと十分に、また均一に混合・混練ができない。

②比表面積もしくは見掛比重（ｇ／ｌ）が大きいため、砥石の成形中に入った空気が成形完了後に取り出す際、膨張してクラックが発生しやすい。

③また仮に成形できたとしても、微細なクラックあるいは欠陥が内在するために加工時に、粒子が凝集・集塊している部分からクラックが発生しやすい。

④さらに上記の問題が解決できたとしても、砥粒の密度が高いために砥石が目づまりして長時間の加工ができない。

この目づまりを解決する方法として連続ドレッシングなどによる強制発刃法もあるが、あらかじめ砥粒が自生発刃するように砥石を製作できればより効果的である。

【０００４】そこで本発明の目的とするところは、クラックの発生を極力防止でき、かつ砥粒の自生作用を活発に発現させつつ、加工生成物の逃げ場を増やし目づまりを未然に防ぐ、高い空孔率をもった超微粉シリカ砥石およびその効果的な製造方法を提供するにある。

【０００５】

【課題を解決するための手段】本発明は上記目的を達成するため次の構成を備える。すなわち、本発明に係る超微粉シリカ砥石は、フェノール樹脂によりコーティングされた超微粉シリカがプレス成形されて空孔率０～８０％に形成されたことを特徴としている。超微粉シリカに対するフェノール樹脂の割合が１～９９vol ％にすると好適である。砥石のビッカース硬さは１～５０Hvであると好適である。また集中度は５０～３００であると好適である。本発明に係る超微粉シリカ砥石の製造方法では、熱硬化性のフェノール樹脂を揮発性の溶媒に溶解した液中に超微粉シリカを加えてスラリー状にし、撹拌して均一に混合する工程と、該混合液体から揮発性成分を飛ばして乾燥させる工程と、乾燥物を粉砕して、超微粉シリカがフェノール樹脂によりコーティングされた粉末状の成形材料を形成する工程と、真空状にした容器内で前記成形材料を金型により所要形状にプレス成形する工程と、得られた成形体を焼成してフェノール樹脂を硬化させる工程とを具備することを特徴としている。金型から成形体を取り出す際に成形体に与圧をかけながら取り出すようにすると成形体にクラックが発生せず、好適である。前記成形材料をプレス成形する際の成形圧力を５０～６０００kgf/cm^2 に設定すると好適である。超微粉シリカに対する熱硬化性のフェノール樹脂の割合を１～９９vol ％にすると好適である。

【０００６】

【作用】本発明に係る超微粉シリカ砥石では、超微粉シリカを熱硬化性フェノール樹脂をバインダとして空孔率０～８０％に成形することによって、シリコンウェハ等の被研削材を目づまりすることなく高精度に鏡面研削することができる。フェノール樹脂の超微粉シリカに対する混合割合を５～１５vol ％にすると、メカノケミカルポリシング後の反応したシリカ砥粒を脱落させる点で好

都合となる。また、ビッカース硬度を１～１０Hvにすると、ソフトタッチな研削が行え、また被加工材が反っている場合に、形状にならった研削が行える点で好都合である。集中度は、５０～１５０程度にすることによって、チップポケットが形成でき、目詰まりを防ぐ点で好都合である。また本発明方法では、成形材料を真空容器中で金型によりプレス成形することによって、クラックや割れを発生させることなく所要形状の砥石に成形できる。特に、金型から成形体を取り出す際、成形体に与圧をかけながら取り出すようにすることでクラックの発生を可及的に少なくできる。

【０００７】

【実施例】以下、本発明の好適な実施例を添付図面に基づいて詳細に説明する。本実施例で用いた超微粉（超微粒子粉末）シリカは、気相法、特に火炎加水分解法により生成されたものであり、現在工業規格に基づいて製造されているシリカ（SiO_2）の中では最も小さい粒子径をもつ高純度の無水シリカ（アエロジル：日本アエロジル株式会社製）のものである。表１は用いた砥粒の種類と仕様を示す。粒径４０ｎｍ（No.OX50）、１６ｎｍ(No.130)、７ｎｍ(No.380)という粒径４０ｎｍ以下の極めて微細な粒子であることがわかる。これら粒子はほぼ球形の１次粒子がふわふわした編目構造を形成し、実際には単独粒子状態では存在しない。ただし、粒径４０ｎｍの場合は例外で、独立した球形の１次粒子がはっきりと見える。

【０００８】一方バインダは、以下の理由で熱硬化性フェノール樹脂を選定した。

①加工途中、水（研削液）に溶けて砥石の形状が崩れない。

②砥粒とバインダを混合し成形した後、熱を加えて硬化できる。

表２は用いたバインダであるPR-50696熱硬化性フェノール樹脂（住友デュレス株式会社製）の仕様を示す。揮発成分が51.7vol ％、165 ℃で63秒経過後ゲル化する液体状態のものである。

【０００９】超微粉シリカ砥石の製作ができなかった原因の中で、粒子の凝集・集塊などによるバインダとの不均一な混合を解決しなければならない。そこで、凝集している粒子にできる限り分散性を与えた液体状態で混合することに着目した。すなわち、図１に示すように、熱硬化性のフェノール樹脂〔各１８ｇ（13.8vol ％）、４０ｇ（26.2vol ％）、８０ｇ（41.5vol ％）、１２４ｇ(52.4vol％)、２０７ｇ(64.7vol％)〕を大量の揮発性溶媒（例えばエチルアルコール）（1200 ｇ）で溶かしたうえ超微粉シリカ粉末１００ｇを加え、スラリー状にした。その後スクリューで約１０分間攪拌してバインダとシリカ粒子が均一になるように混合した。その際、異物などが混入しないように十分注意した。

【００１０】その後、超微粉シリカ、フェノール樹脂、エチルアルコールの混合液体状態から揮発成分を飛ばして無くすため、真空乾燥機により５０℃、２時間程度乾燥させた。乾燥された混合粉末は、ふわふわした混合前のシリカ粉末とは全く異なり、かさかさとした状態で軽く固まっている。これを乳鉢の中で粉砕して粉末状にした。フェノール樹脂はシリカ粒子の周囲にほぼ均一にコーティングされた状態となっている。このようにして成形材料を調整できた。

【００１１】図２は成形装置１０の概要を示す。１２は本体容器であり、内部状態が外部から見えるように透明なアクリル樹脂等で形成すると好適である。本体容器１２下面側は底板１４により取り外し自在に密閉される。１６はOリングである。また１８はプレス装置のロッド２０により押圧されて本体容器１２内を移動可能な可動盤である。可動盤１８はロッド２０下端に固定してもよい。１９はOリングである。２１は超硬合金からなる金型であり、ダイス２２、下パンチ２８、上パンチ３０を有する。ダイス２２には上下方向に貫通する貫通孔２４が形成され、底板１４上に配置されたスプリング２６上にバランスをとって載置可能になっている。下パンチ２８は底板１４上に配置され、上部が貫通孔２４に下方から摺入している。上パンチ３０は下部が貫通孔２４に上方から嵌入する。上パンチ３０は可動盤１８下面に固定しておいてもよい。下パンチ２８上端面と上パンチ３０下端面との間にキャビティ３２が形成される。

【００１２】底板１４には本体容器１２と外部とを連通する吸引孔３４が設けられており、該吸引孔３４にはホースを介して図示しない真空ポンプが接続されて、本体容器１２内の空気を排除することにより本体容器１２内を真空状態にできる。上パンチ３０は成形品をダイス２２から突き出すためにダイス２２の高さよりも長い寸法に形成されている。図３は与圧装置３６を示す。この与圧装置３６は、成形後、本体容器１２から取り出され、上パンチ３０が下側にくるようにして適宜な台上に載置された金型２１のダイス２２上面側に当接する複数本の押圧ロッド３８と、押圧ロッド３８上端側を連結する押圧板４０と、押圧板４０下面と下パンチ２８との間に配置される与圧スプリング４２を有する。４４はショックアブソーバである。

【００１３】上記成形装置１０を用いて次のように砥石の成形を行うことができる。まず成形装置１０において、可動盤１８と上パンチ３０とは成形装置から外されている。上記のようにしてバインダと超微粉シリカとが混合され、粉末状に調整された成形材料を、開放されたキャビティ３２内に所定量入れる。次に上パンチ３０を貫通孔２４に挿入した後、できる限り上パンチ３０の上面に近い位置まで可動盤１８を移動して本体容器１２を密閉する。その後真空ポンプで引いて本体容器１２内を真空状態とし、次いでプレス装置により可動盤１８、上パンチ３０を介して成形材料を加圧し、成形する。成形

材料中の空気は真空ポンプにより吸引されているから成形材料中に空気が巻き込まれることなく成形される。

【００１４】成形完了後、金型２１を本体容器１２内から取り出す。取り出した金型２１は上パンチ３０が下にくるように適宜な台上に載置し、図３に示すように、与圧装置３６をセットする。押圧板４０に適宜な加圧装置により荷重を加え、押圧板４０を押す。これにより押圧ロッド３８を介してダイス２２が下方に押され、成形体４６は上パンチ３０により突き出されてくる。その際成形体４６には与圧スプリング４２により圧力が加わる。すなわち与圧をかけながら成形体４６をダイス２２から取り出すのである。このように与圧をかけながら取り出すことにより、欠けのない良好な成形品が得られた。なお、与圧をかけないで成形体４６を上パンチ３０により突き出すと、成形体４６が貫通孔２４から抜け出るときに貫通孔２４から抜け出る側が急に圧力フリー状態となることから、成形体４６に割れ、欠けなどが生じた。得られた成形体４６を真空乾燥器を用い180 ℃、約１時間焼成してフェノール樹脂を硬化させて砥石を得た。

【００１５】上記成形装置１０で真空に引きながら、500Kgf/cm^{2}で加圧成形した場合の各成形体の密度の差異を図４に示す。混合粉末の真比重は、超微細シリカ粉末にフェノール樹脂を加える割合により表３に示すように、もとのシリカ粉末のみの真比重とは異なる値を有する。空孔率は次式により計算される。

(1) シリカ粉末のみの空孔率＝（シリカ粉末の真比重－成形体の密度）／シリカ粉末の真比重

(2) 混合シリカ粉末の空孔率＝（混合シリカ粉末の真比重－成形体の密度）／混合シリカ粉末の真比重

上記の式に基づいて計算した混合シリカ粉末の空孔率を図５に示した。

【００１６】図４、図５から以下のことが確認、もしくは推測される。

①混合シリカ粉末は、混合前の粉末と比べてややバラツキはあるが、全体的に密度および空孔率ともに低い傾向を示す。これはバインダとして用いたフェノール樹脂の真比重が１g/cm^3 程度で、比較的低いためであると思われる。

②空孔率の算出については混合前のシリカ粉末の場合、真比重が2.2g/cm^{3}であったが、フェノール樹脂の体積割合が多くなるほど、混合シリカ粉末の真比重は低くなり、フェノール樹脂の体積割合が41.5vol %の場合には1.79g/cm^3 まで下がる。

③しかし混合前のシリカ粉末は、嵩比重が約60g/l という非常にふわふわとした状態となっているため、フェノール樹脂の体積割合が増加することによって混合シリカ粉末の嵩比重は高くなると考えられる。また、結合度も高くなり、成形体の密度が高くなると予測される。

④粒径が大きくなるほど比表面積が小さくなるため密度は高くなるが、粒径がより小さい７ｎｍ、１６ｎｍの場合は、各者とも大差はなかった。しかし、比較的粒径が大きい４０ｎｍの場合、密度が１g/cm^3 程度まで高くなった。

⑤混合シリカ粉末の空孔率は、成形体の密度特性とは反対の傾向を有する。すなわち、フェノール樹脂の体積割合が増加するほど、粒径が大きくなるほど、空孔率は低くなるが、全体的に50vol %前後を維持している。

【００１７】次に、図６は、粒径４０ｎｍの超微細シリカ粉末No. OX50 に各割合でフェノール樹脂をコーティングした場合の、成形圧力に対する空孔率特性を示す。バインダをコーティングしないものと比較して成形圧力の増加、バインダ量の増加に応じて空孔が少なくなっている。空孔率の分布は10～70vol %という非常に広い範囲にわたっている。図７に、砥粒の集中度と砥粒率を空孔率から換算して示す。砥石の空孔率および集中度特性より、バインダの多いものは、成形圧力に応じ空孔を少なくすることは可能であるが、その分集中度の増加割合は少ないものとなっている。バインダ量13.8vol %の場合、成形圧力が1000kgf/cm^3 を越える領域ではバインダをコーティングしないものより砥粒の集中度がわずかに上回っている。

【００１８】図８に砥石の硬さをマイクロビッカース硬度計により、荷重200gで測定した結果を示す。砥石の硬さ、すなわち結合度は、成形圧力に応じてHv５～２８の間に自由にコントロールできた。成形圧力が500kgf/cm^{2}の低い場合では、バインダ量が30vol %を越える範囲で硬さが逆に減少している。これらに示すように、成形体（砥石）はバインダ量および成形圧力を調整することにより所望の集中度、空孔率および結合度の砥石を製作できるという特徴をもつ。ちなみに、粒径４０ｎｍのシリカ粒子（OX50）の場合、バインダがコーティングしてあれば、真空中ではなくても通常のプレス成形法で砥石が成形できた。

【００１９】次に超微粉シリカ砥石を製作し、成形条件と砥石の研削特性を調べた。砥粒は平均粒径４０ｎｍという比較的粗いNo. OX50 にフェノール樹脂を加えて混合した後、成形・焼成したものを用い、砥石外径150 mm、幅10ｍｍのカップ型のものを作成し、加工前において砥石の形状精度を向上させるため、粒度＃1200のダイヤモンド砥石でツルーイングを行った。超微粉シリカ砥石の鏡面研削への適用は、まずインフィールド研削として粒径にほぼ制限をもたない定圧切込研削に近い方式により行った。被加工物であるシリコンウェハの鏡面状態を加工中でも確認できるよう、片面ラップ盤（ラポテスターMG773B型、株式会社マルトー製）の定盤に被削材を、ワーク軸に砥石を取り付けたあと両方とも回転させた。この際、砥石は常に被削材に一定の圧力をかけながら、左右にオシレート運動をする。被削材は直径３～６インチ生シリコンウェハを粒度＃1200のダイヤモンド砥石により、機上で平面出し後、鏡面加工を施した。

【0020】シリコンウェハの鏡面研削特性として、面粗さについては非接触式表面粗さ計（WYKO:TOPO-2D、WYKO社製）で、評価長さ324 μｍ、波長647.7 ｎｍにより測定を行い、研削比については３点指示マイクロメータで評価を行った。超微粉砥石による鏡面研削では、チップポケットが少なく目づまりが生じ易い。そこでこのような目詰まりが起こらないようにするため、チップポケット数を増加させて、できる限り多孔質状態のものを適用した。図９に比較的高い空孔率である45〜55vol ％の範囲にある砥石により得られた仕上げ面粗さと研削比を示す。空孔率が高くなるほど仕上げ面粗さは向上するが、空孔率50vol ％以上では大きな変化はなくRa１ｎｍ程度を維持した。また空孔率50vol ％前後までは研削比が１に近づくが、それ以上高くすると徐々に減少した。これは空孔率をある程度高くすることで、加工による反応生成物の除去が容易になった状態と考えられ、ほぼ同一な結合度の場合、砥粒の集中度の高い方が除去能率も高く、加工面粗さも向上させる効果が生ずるものと思われる。

【0021】図１０は、本実施例の超微粉シリカ砥石の結合度が鏡面研削特性に及ぼす影響を示す。比較的高い空孔率と砥粒集中度を有すると予想されるバインダの体積割合7 〜19vol ％範囲では、結合度が低くなるほど加工面粗さと研削比が高くなった。この範囲より高い結合度のシリカ砥石を適用してみたが、硬く目づまりが生じ、長時間の加工が困難であった。したがって本実施例の超微粉シリカ砥石は高空孔率・高集中度・低結合度の方が、シリコンウェハの鏡面研削特性がより好適なものになると言える。

【0022】

【表１】

ＮＯ．	Ｏ×５０	１３０	３８０
表　面　積（m^2/g）	５０±１５	１２０±２０	３８０±３０
粒子サイズ（nm）	４０	１６	７
見掛け比重（g/l）	８０	６０	６０
真　比　重（g/cm^3）	２．２		

【0023】

【表２】

比重　（g/l）	１．０２６
粘　度（poise）	０．２
ゲル化温度時間	１６５℃，６３ｓｅｃ
不揮発性成分（vol％）	４８．３

【0024】

【表３】

ﾊﾞｲﾝﾀﾞｰ量（vol％）	ｼﾘｶ微粉末の重量（g）	ﾊﾞｲﾝﾀﾞｰ重量（g）	混合物の真重量（g/cm^3）
0	−	−	2.20
13.8	50	9	2.06
26.2	50	20	1.94
41.5	50	40	1.79

【0025】

【発明の効果】本発明に係る超微粉シリカ砥石では、超微粉シリカを熱硬化性フェノール樹脂をバインダとして空孔率０〜８０％、特に５０〜８０％に成形することによって、シリコンウェハ等の被研削材を目づまりすることなく高精度に鏡面研削することができる。フェノール樹脂の超微粉シリカに対する混合割合を１〜９９vol ％、特に５〜１５vol ％にすると、反応シリカ砥粒の脱落で目詰まりが防げる点で好都合となる。また、ビッカース硬度を１〜４０Hv、特に１〜１０Hvにすると、ソフトタッチな研削が行え、被加工材の形状にならった研削が行える点で好都合である。集中度は、５０〜３００、特に５０〜１５０程度にすることによって、チップポケットが形成でき、目詰まりを防ぐ点で好都合である。また本発明方法では、成形材料を真空容器中で金型によりプレス成形することによって、クラックや割れを発生させることなく所要形状の砥石に成形できる。特に、金型から成形体を取り出す際、成形体に与圧をかけながら取り出すようにすることでクラックの発生を可及的に少なくできる。

【図面の簡単な説明】

【図１】砥石の製作工程図である。

【図２】成形装置の概略を示す断面図である。

【図３】与圧装置の概略を示す説明図である。

【図４】500kgf/cm^2 で加圧成形した場合の成形体の密度を示すグラフである。

【図５】500kgf/cm^2 で加圧成形した場合の成形体の空孔率を示すグラフである。

【図６】成形圧力に対する空孔率特性を示すグラフである。

【図７】砥粒の集中度と砥粒率を示すグラフである。

【図８】砥石の結合度（硬さ）を示すグラフである。

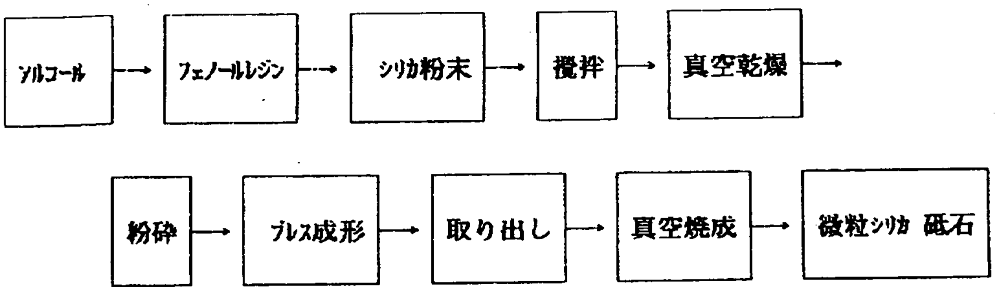
【図９】仕上げ面粗さと研削比を示すグラフである。

【図１０】砥石の結合度が鏡面研削特性に及ぼす影響を示すグラフである。

【符号の説明】

１０	成形装置
＊１２	本体容器
１４	底板
１８	可動盤
２１	金型
２２	ダイス
２４	貫通孔
２６	スプリング
２８	下パンチ
３０	上パンチ
３２	キャビティ
３６	与圧装置
３８	押圧ロッド
４０	押圧板
４２	与圧スプリング
＊４６	成形体

【図１】



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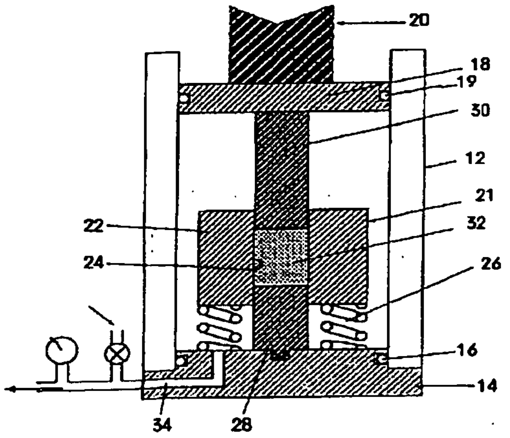

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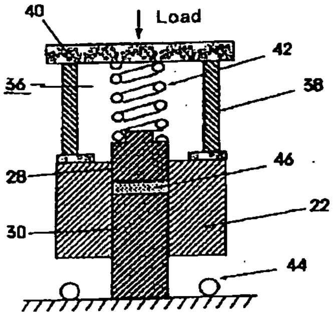

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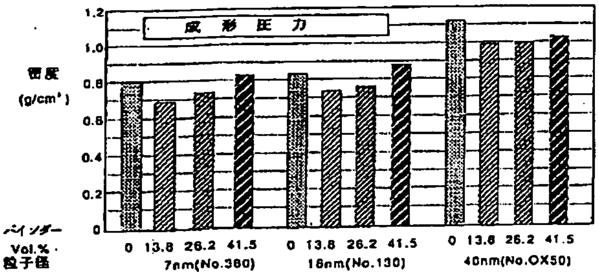

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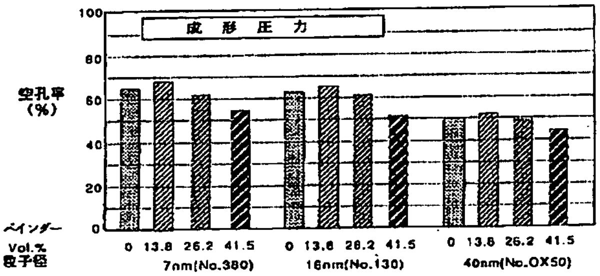

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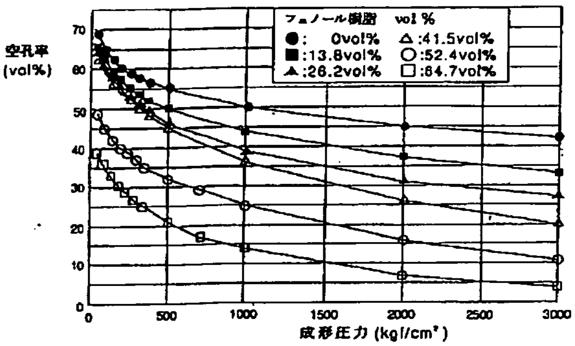

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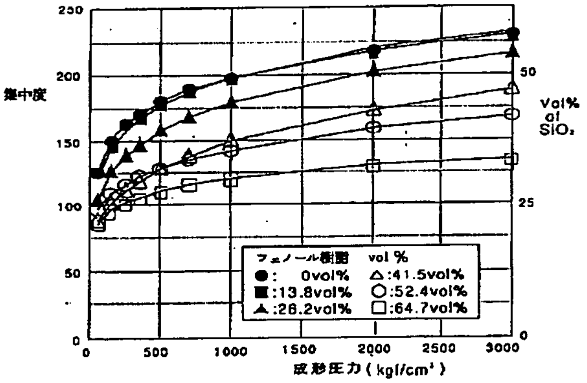

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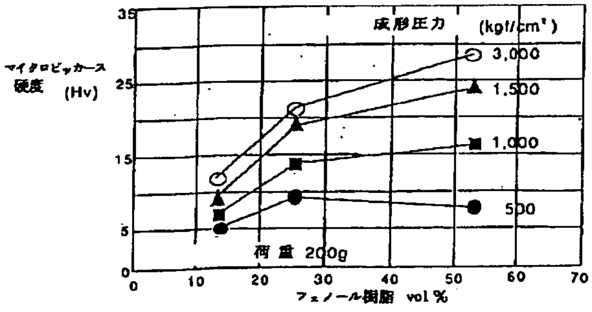
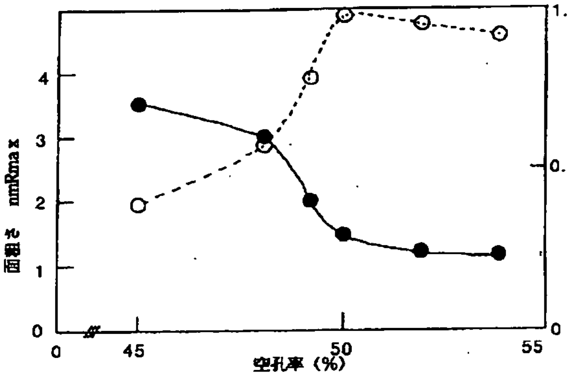
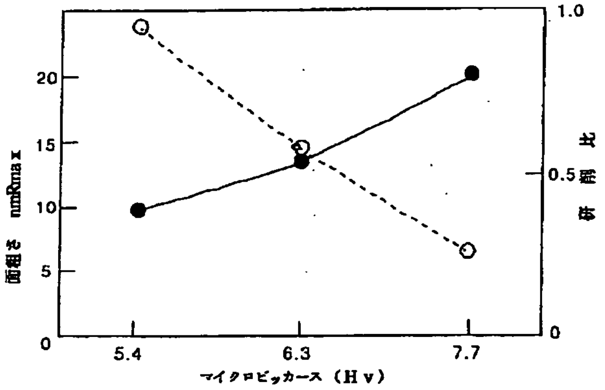


【図９】



【図１０】



フロントページの続き

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JAPANESE [JP,09-047969,A]

CLAIMS DETAILED DESCRIPTION TECHNICAL FIELD PRIOR ART EFFECT OF THE INVENTION MEANS OPERATION EXAMPLE DESCRIPTION OF DRAWINGS DRAWINGS

[Translation done.]

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CLAIMS

[Claim(s)]
[Claim 1] The superfines silica grinding stone characterized by having carried out press forming of the superfines silica coating was carried out [the silica] by phenol resin, and forming it in 0 - 80% of void contents.
[Claim 2] The superfines silica grinding stone according to claim 1 characterized by the percentage of phenol resin to a superfines silica being 1 - 99vol%.
[Claim 3] The superfines silica grinding stone according to claim 1 or 2 characterized by Vickers hardness number being 1-40Hv.
[Claim 4] The superfines silica grinding stone according to claim 1, 2, or 3 characterized by degree of concentration being 50-300.
[Claim 5] The process which adds a superfines silica into the liquid which dissolved thermosetting phenol resin in the volatile solvent, makes the shape of a slurry, is stirred and is mixed to homogeneity, The process which flies a volatile component and is dried from this mixed liquor object, and a dry matter are ground. The process which forms the powder-like molding material with which coating of the superfines silica was carried out by phenol resin, The manufacture approach of the superfines silica grinding stone characterized by providing the process which carries out press forming of said molding material to a necessary configuration with metal mold within the container made into the shape of a vacuum, and the process which the acquired Plastic solid is calcinated [process] and stiffens phenol resin.
[Claim 6] The manufacture approach of the superfines silica grinding stone according to claim 5 characterized by taking out applying pressurization to a Plastic solid in case a Plastic solid is picked out from metal mold.
[Claim 7] the compacting pressure at the time of carrying out press forming of said molding material -- 10 - 6000 kgf/cm2 it is -- the manufacture approach of the superfines silica grinding stone according to claim 5 or 6 characterized by things.
[Claim 8] The manufacture approach of the superfines silica grinding stone according to claim 5, 6, or 7 characterized by the rate of thermosetting phenol resin to a superfines silica being 1-99vol %.

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DETAILED DESCRIPTION

[Detailed Description of the Invention]
[0001]
[Industrial Application] This invention relates to a superfines silica grinding stone and its manufacture approach.
[0002]
[Background of the Invention] Mirror plane finishing precision with an advanced semi-conductor wafer is required. In order to obtain the machined surface granularity of nanometer order, it is an effective means to perform grinding using the grinding stone which consists of a detailed abrasive grain. Although development of the grinding stone which fixed the detailed abrasive grain with resin binders, such as phenol resin, is performed in order to realize this, the trouble has not yet realized many manufactures of the grinding stone using the superfines (ultrafine particle powder) whose diameters of an abrasive grain are a number - 10nm of numbers.
[0003] The following causes are cited as a reason which the grinding stone using the abrasive grain of superfines does not realize.
** since it is condensation and conglomerate of the particle which is one of the descriptions which ultrafine particle powder has -- a binder -- enough -- moreover, mixing and kneading are not made to homogeneity.
** Since specific surface area or bulk specific gravity (g/l) is large, in case the air which entered during shaping of a grinding stone takes out after the completion of shaping, expand and it is easy to generate a crack.
** Even if it is able to fabricate again, since a detailed crack or a detailed defect is inherent, a particle will tend to generate a crack from condensation and the part which is carrying out the conglomerate at the time of processing.
** Even if the further above-mentioned problem is solvable, since the consistency of an abrasive grain is high, a grinding stone carries out loading and processing of long duration cannot be performed.
Although there is also a cutting-edge method from compulsion by a continuation dressing etc. as an approach of solving this loading, it is more effective, if a grinding stone can be manufactured so that an abrasive grain may carry out the cutting edge from spontaneous generation beforehand.
[0004] Then, the place made into the purpose of this invention is to offer the superfines silica grinding stone with a high void content which increases the refuge of a processing product and prevents loading, and its effective manufacture approach, being able to prevent generating of a crack as much as possible, and making a spontaneous generation operation of an abrasive grain discover actively.
[0005]
[Means for Solving the Problem] This invention is equipped with the next configuration in order to attain the above-mentioned purpose. That is, the superfines silica grinding stone concerning this invention is characterized by having carried out press forming of the superfines silica coating was carried out [the silica] by phenol resin, and forming it in 0 - 80% of void contents. It is suitable if the rate of phenol resin to a superfines silica makes it 1 - 99vol %. The Vickers

hardness number of a grinding stone is suitable in it being 1-50Hv. Moreover, degree of concentration is suitable in it being 50-300. By the manufacture approach of the superfines silica grinding stone concerning this invention The process which adds a superfines silica into the liquid which dissolved thermosetting phenol resin in the volatile solvent, makes the shape of a slurry, is stirred and is mixed to homogeneity, The process which flies a volatile component and is dried from this mixed liquor object, and a dry matter are ground. The process which forms the powder-like molding material with which coating of the superfines silica was carried out by phenol resin, It is characterized by providing the process which carries out press forming of said molding material to a necessary configuration with metal mold within the container made into the shape of a vacuum, and the process which the acquired Plastic solid is calcinated [process] and stiffens phenol resin. If it is made to take out, applying pressurization to a Plastic solid in case a Plastic solid is picked out from metal mold, a crack does not occur in a Plastic solid but it is suitable for it. It is the compacting pressure at the time of carrying out press forming of said molding material 50 - 6000 kgf/cm2 It is suitable if it sets up. It is suitable if the rate of thermosetting phenol resin to a superfines silica is made into 1 - 99vol %.

[0006]

[Function] Mirror grinding can be carried out with high precision by fabricating a superfines silica in the superfines silica grinding stone concerning this invention to 0 - 80% of void contents by using thermosetting phenol resin as a binder, without carrying out loading of the abrasives-ed, such as a silicon wafer. If the mixed rate to the superfines silica of phenol resin is made into 5 - 15vol %, it will become convenient in that the silica abrasive grain to which it reacted after mechanochemical polishing is omitted. Moreover, if Vickers hardness is set to 1-10Hv, when grinding [****** / soft] can be performed and the work material has curved, it is convenient at the point that grinding learned from the configuration can be performed. Degree of concentration is convenient by carrying out to 50 to about 150 at the point which can form a chip pocket and prevents blinding. Moreover, it can fabricate by this invention approach to the grinding stone of a necessary configuration by carrying out press forming of the molding material with metal mold in a vacuum housing, without generating a crack and a crack. In case a Plastic solid is especially picked out from metal mold, generating of a crack can be lessened as much as possible by making it take out, applying pressurization to a Plastic solid.

[0007]

[Example] Hereafter, the suitable example of this invention is explained to a detail based on an accompanying drawing. The superfines (ultrafine particle powder) silica used by this example is the thing of the anhydrous silica (Aerosil: product made from Japanese Aerosil, Inc.) with the smallest particle diameter in the silica (SiO2) which is generated by a gaseous-phase method, especially flame hydrolysis, and is manufactured based on current British Standard of a high grade. Table 1 shows the class and specification of the used abrasive grain. It turns out that it is a very detailed particle with a particle size [of the particle size of 40nm (No.OX50), 16nm (No.130), and 7nm (No.380)] of 40nm or less. These particles form the network structure which the almost globular form primary particle had lightly, and do not exist in the state of an independent particle in fact. However, in the case of 40nm particle size, it is an exception, and it can be seen clearly. [of the independent globular form primary particle]

[0008] On the other hand, the binder selected thermosetting phenol resin by the following reasons.

** In the middle of processing, melt into water (grinding fluid) and the configuration of a grinding stone does not collapse.

** Heat can be applied and hardened after mixing and fabricating an abrasive grain and a binder. Table 2 shows the specification of the PR-50696 thermosetting phenol resin (product made from Sumitomo DEYURESU, Inc.) which is the used binder. A volatile component is the thing of the liquid condition gelled after 63-second progress by 51.7vol(s) % and 165 **.

[0009] In the cause whose manufacture of a superfines silica grinding stone was not completed, uneven mixing with the binder by condensation, conglomerate, etc. of a particle must be solved. Then, it noted mixing in the state of the liquid which gave dispersibility to the condensed particle as much as possible. That is, as shown in drawing 1 , after melting thermosetting phenol resin

[18g each (13.8vol %), 40g (26.2vol %), 80g (41.5vol %), and 124g (52.4vol%), 207g (64.7vol%)] by a lot of volatile solvents (for example, ethyl alcohol) (1200 g), 100g of superfines silica powder was added, and it was made the shape of a slurry. It mixed so that it might stir for about 10 minutes with a screw after that and a binder and a silica particle might become homogeneity. It took care enough that a foreign matter etc. did not mix at that time.

[0010] Then, in order to fly and lose a volatile component from the mixed liquor object condition of a superfines silica, phenol resin, and ethyl alcohol, 50 degrees C was dried with the vacuum dryer for about 2 hours. Completely unlike the silica powder before mixing carried out lightly, the dried mixed powder has become hard lightly in the condition of having carried out rustlingly. This was ground in the mortar and it was made the shape of powder. Phenol resin is in the condition that coating was mostly carried out to homogeneity around the silica particle. Thus, the molding material has been adjusted.

[0011] Drawing 2 shows the outline of shaping equipment 10. 12 is a body container, and if it is formed with transparent acrylic resin etc. so that an internal state may be in sight from the outside, it is suitable. A body container 12 inferior-surface-of-tongue side is sealed by the bottom plate 14 free [removal]. 16 is an O ring. Moreover, 18 is pressed with the rod 20 of press equipment, and is a movable movable head about the inside of the body container 12. A movable head 18 may be fixed to rod 20 lower limit. 19 is an O ring. 21 is metal mold which consists of cemented carbide, and has a dice 22, bottom punch 28, and upper punch 30. The through tube 24 penetrated in the vertical direction is formed in a dice 22, on the spring 26 arranged on a bottom plate 14, balance is maintained and installation has become possible. Bottom punch 28 is arranged on a bottom plate 14, and the upper part is carrying out ** ON to the through tube 24 from the lower part. The lower part inserts upper punch 30 in a through tube 24 from the upper part. Upper punch 30 may be fixed to movable head 18 inferior surface of tongue. A cavity 32 is formed between a bottom punch 28 upper-limit side and an upper punch 30 lower-limit side.

[0012] The suction hole 34 which opens the body container 12 and the exterior for free passage is formed in the bottom plate 14, the vacuum pump which is not illustrated through a hose is connected to this suction hole 34, and the inside of the body container 12 is made to a vacua by eliminating the air in the body container 12. Upper punch 30 is formed in the dimension longer than the height of a dice 22 in order to project mold goods from a dice 22. Drawing 3 shows pressurization equipment 36. the pressurization arranged between two or more press rods 38 which contact the dice 22 top-face side of the metal mold 21 laid on the proper base as this pressurization equipment 36 was picked out from the body container 12 after shaping and upper punch 30 came to the bottom, the press plate 40 which connects a press rod 38 upper-limit side, and press plate 40 inferior surface of tongue and bottom punch 28 — it has a spring 42. 44 is a shock absorber.

[0013] A grinding stone can be fabricated as follows using the above-mentioned shaping equipment 10. In shaping equipment 10, a movable head 18 and upper punch 30 are first removed from shaping equipment. It is specified quantity ON **** in the cavity 32 wide opened in the molding material which the binder and the superfines silica were mixed as mentioned above, and was adjusted in the shape of powder. Next, after inserting upper punch 30 in a through tube 24, a movable head 18 is moved to the location near the top face of upper punch 30 as much as possible, and the body container 12 is sealed. It lengthens with a vacuum pump after that, and the inside of the body container 12 is made into a vacua, and subsequently a molding material is pressurized through a movable head 18 and upper punch 30 with press equipment, and it fabricates. The air in a molding material is fabricated without involving in air into a molding material, since it is drawn in by the vacuum pump.

[0014] Metal mold 21 is taken out from the inside of the body container 12 after the completion of shaping. The taken-out metal mold 21 is laid on a proper base so that upper punch 30 may come downward, and as shown in drawing 3 , it sets pressurization equipment 36. A load is added to the press plate 40 with a proper pressurizer, and the press plate 40 is pushed. Thereby, through the press rod 38, a dice 22 is pushed caudad and Plastic solid 46 is projected by upper punch 30. that time — Plastic solid 46 — pressurization — a pressure is added with a spring 42.

That is, Plastic solid 46 is picked out from a dice 22, applying pressurization. Thus, good mold goods without a chip were obtained by taking out applying pressurization. In addition, since the side which escapes from and comes out of a through tube 24 was pressure free suddenly when Plastic solid 46 was projected by upper punch 30 without applying pressurization, and Plastic solid 46 escaped from and came out of a through tube 24, it was divided into Plastic solid 46 and the chip etc. arose. acquired Plastic solid 46 — a vacuum dryer — using — 180 ** — it calcinated for about 1 hour, phenol resin was stiffened, and the grinding stone was obtained.
[0015] The difference in the consistency of each Plastic solid at the time of carrying out pressing by 500 Kgf/cm2 is shown in drawing 4 , lengthening to a vacuum with the above-mentioned shaping equipment 10. The true specific gravity of mixed powder has a different value from the true specific gravity of only the silica powder of a basis, as the rate of overly adding phenol resin to detailed silica powder shows to Table 3. A void content is calculated by the degree type.
(1) The void content of only silica powder = (consistency of the true-specific-gravity-Plastic solid of silica powder) true specific gravity of /silica powder (2) The void content of mixed silica powder = (consistency of the true-specific-gravity-Plastic solid of mixed silica powder) the void content of the mixed silica powder calculated based on the formula of the true-specific-gravity above of /mixing silica powder was shown in drawing 5 .
[0016] The following things are checked or guessed from drawing 4 and drawing 5 .
** Although there is variation a little compared with the powder before mixing, on the whole, as for mixed silica powder, a consistency and a void content show a low inclination. For this, the true specific gravity of the phenol resin used as a binder is 1 g/cm3. With extent, it is thought that it is because it is comparatively low.
** the case where the true specific gravity of mixed silica powder becomes low, and the volume rate of phenol resin is 41.5vol(s) %, so that true specific gravity was 2.2 g/cm3 in the case of the silica powder before mixing, but the volume rate of phenol resin increases about calculation of a void content — 1.79 g/cm3 up to — it falls.
** however, the silica powder before mixing — relative bulk density — about 60 g/l ** — since it is in the condition to say of having made it extraordinary lightly, when the volume rate of phenol resin increases, it is thought that the relative bulk density of mixed silica powder becomes high. Moreover, degree of coupling also becomes high and the consistency of a Plastic solid is predicted to become high.
** Since specific surface area became small so that particle size becomes large, the consistency became high, but when it was 7nm with a smaller particle size, and 16nm, there was no great difference in each **. However, when it is 40nm with a comparatively large particle size, a consistency is 1 g/cm3. It became high to extent.
** The void content of mixed silica powder has an inclination opposite to the consistency property of a Plastic solid. That is, although a void content becomes low so that the volume rate of phenol resin increases, and particle size becomes large, on the whole, 50vol(s) % order is maintained.
[0017] next, drawing 6 — the particle size of 40nm — overly detailed — silica powder No.OX50 The void content property over the compacting pressure at the time of coating phenol resin with each rate is shown. As compared with what does not coat a binder, the hole has decreased according to the increment in compacting pressure, and the increment in the amount of binders. Distribution of a void content is crossed to the very large range 10-70vol %. The degree of concentration and the grain ratio of an abrasive grain are converted and shown in drawing 7 from a void content. Although it is possible for what has more binders than the void content and degree-of-concentration property of a grinding stone to lessen a hole according to compacting pressure, the increment rates of among the recentralization whenever are few things. In amount of binders 13.8vol %, compacting pressure is 1000 kgf/cm3. In the field to cross, the degree of concentration of an abrasive grain has exceeded slightly from what does not coat a binder.
[0018] The result of having measured the hardness of a grinding stone by 200g of loads with the micro Vickers hardness meter to drawing 8 is shown. According to compacting pressure, the hardness of a grinding stone, i.e., degree of coupling, was freely controllable between Hv(s) 5-28.

Hardness is decreasing conversely in the range in which the amount of binders exceeds [compacting pressure] 30vol(s) % by the case of 500 kgf/cm2 of being low. As shown in these, a Plastic solid (grinding stone) has the description that the grinding stone of desired degree of concentration, a void content, and degree of coupling can be manufactured, by adjusting the amount of binders, and compacting pressure. incidentally — silica particle (OX50) with a particle size of 40nm a case — a binder — coating — **** — if — the inside of a vacuum — it is not — even if — usual — the grinding stone has been fabricated by the press-forming method.
[0019] Next, the superfines silica grinding stone was manufactured and the process condition and the grinding property of a grinding stone were investigated. An abrasive grain is comparatively coarse No.OX50 called the mean particle diameter of 40nm. After adding phenol resin and mixing, in order to create the thing of grinding stone outer-diameter 150 mm and a cup mold with a width of face of 10mm and to raise the configuration precision of a grinding stone before processing using what was fabricated and calcinated, truing was performed by the diamond wheel of grain-size #1200. The method near the constant-pressure cut grinding which does not have a limit in particle size mostly as infield grinding probably performed application to the mirror grinding of a superfines silica grinding stone. After attaching **-ed material in the surface plate of a single side lapping machine (a RAPOTE star MG773B mold, Maruto Make) and attaching a grinding stone in a work-piece shaft so that the mirror plane condition of the silicon wafer which is a workpiece can be checked also in processing, both made it rotate. Under the present circumstances, a grinding stone carries out oscillation movement to right and left, always putting a fixed pressure on **-ed material. By the diamond wheel of grain-size #1200, **-ed material was on board in the diameter student silicon wafer of 3-6 inches, and performed mirror plane processing after Taira figuring.
[0020] As a mirror grinding property of a silicon wafer, about field coarseness, it is a non-contact type surface roughness meter (WYKO:TOPO-2D, product made from WYKO), and is the evaluation die length 324. It measured by mum and wavelength 647.7 nm, and a three-point indicating micrometer estimated the grinding ratio. In the mirror grinding by the superfines grinding stone, there are few chip pockets and it is easy to produce loading. Then, in order to make it such blinding not happen, the number of chip pockets was made to increase and the thing of a porosity condition was applied as much as possible. The machined surface granularity and the grinding ratio which were obtained by the grinding stone shown in drawing 9 in the range of 45 which is a comparatively high void content - 55vol % are shown. Although machined surface granularity improved so that the void content became high, above void content 50vol %, there is no big change and about Ra1nm was maintained. Moreover, although the grinding ratio approached 1 up to void content 50vol % order, when it was made high more than it, it decreased gradually. This is making a void content to some extent high, and is considered to be the condition that removal of the resultant by processing became easy, when it is the almost same degree of coupling, removal efficiency also has the high one where the degree of concentration of an abrasive grain is higher, and it is thought that the effectiveness of also making surface roughness improving arises.
[0021] Drawing 10 shows the effect the degree of coupling of the superfines silica grinding stone of this example affects a mirror grinding property. In the volume rate 7 of a binder - 19vol % range which are expected to have a comparatively high void content and abrasive grain degree of concentration, surface roughness and a grinding ratio became high, so that degree of coupling became low. Although the silica grinding stone of degree of coupling higher than this range was applied, loading arose firmly and processing of long duration was difficult. Therefore, it can be said that the superfines silica grinding stone of this example becomes what has the one of a high void content, quantity degree of concentration, and low degree of coupling more suitable [the mirror grinding property of a silicon wafer].
[0022]
[Table 1]

ＮＯ．	Ｏ×５０	１３０	３８０
表面積 (m^2/g)	５０±１５	１２０±２０	３８０±３０
粒子サイズ (nm)	４０	１６	７
見掛け比重 (g/l)	８０	６０	６０
真比重 (g/cm^3)	２．２		

[0023]
[Table 2]

比重 (g/l)	１．０２６
粘度 (poise)	０．２
ゲル化温度時間	１６５℃，６３ｓｅｃ
不揮発性成分 (vol%)	４８．３

[0024]
[Table 3]

バインダー量 (vol%)	シリカ微粉末の重量 (g)	バインダー重量 (g)	混合物の真重量 (g/cm^3)
0	−	−	2.20
13.8	50	9	2.06
26.2	50	20	1.94
41.5	50	40	1.79

[0025]
[Effect of the Invention] In the superfines silica grinding stone concerning this invention, mirror grinding can be carried out with high precision by fabricating a superfines silica to 50 - 80% especially 0 - 80% of void contents by using thermosetting phenol resin as a binder, without carrying out loading of the abrasives-ed, such as a silicon wafer. About the mixed rate to the superfines silica of phenol resin, if it is especially made 5 - 15vol %, it will become convenient at 1-99vol % and the point which can prevent blinding by omission of a reaction silica abrasive grain. Moreover, if Vickers hardness is especially set to 1-10Hv one to 40 Hv, it is convenient at the point that grinding [****** / soft] can be performed and grinding learned from the configuration of a work material can be performed. Degree of concentration is convenient at the point which can form a chip pocket and prevents blinding 50-300, and by carrying out to 50 to about 150 especially. Moreover, it can fabricate by this invention approach to the grinding stone of a necessary configuration by carrying out press forming of the molding material with metal mold in a vacuum housing, without generating a crack and a crack. In case a Plastic solid is especially picked out from metal mold, generating of a crack can be lessened as much as possible by making it take out, applying pressurization to a Plastic solid.

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TECHNICAL FIELD

[Industrial Application] This invention relates to a superfines silica grinding stone and its manufacture approach.

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PRIOR ART

[Background of the Invention] Mirror plane finishing precision with an advanced semi-conductor wafer is required. In order to obtain the machined surface granularity of nanometer order, it is an effective means to perform grinding using the grinding stone which consists of a detailed abrasive grain. Although development of the grinding stone which fixed the detailed abrasive grain with resin binders, such as phenol resin, is performed in order to realize this, the trouble has not yet realized many manufactures of the grinding stone using the superfines (ultrafine particle powder) whose diameters of an abrasive grain are a number - 10nm of numbers.
[0003] The following causes are cited as a reason which the grinding stone using the abrasive grain of superfines does not realize.
** since it is condensation and conglomerate of the particle which is one of the descriptions which ultrafine particle powder has -- a binder -- enough -- moreover, mixing and kneading are not made to homogeneity.
** Since specific surface area or bulk specific gravity (g/l) is large, in case the air which entered during shaping of a grinding stone takes out after the completion of shaping, expand and it is easy to generate a crack.
** Even if it is able to fabricate again, since a detailed crack or a detailed defect is inherent, a particle will tend to generate a crack from condensation and the part which is carrying out the conglomerate at the time of processing.
** Even if the further above-mentioned problem is solvable, since the consistency of an abrasive grain is high, a grinding stone carries out loading and processing of long duration cannot be performed.
Although there is also a cutting-edge method from compulsion by a continuation dressing etc. as an approach of solving this loading, it is more effective, if a grinding stone can be manufactured so that an abrasive grain may carry out the cutting edge from spontaneous generation beforehand.
[0004] Then, the place made into the purpose of this invention is to offer the superfines silica grinding stone with a high void content which increases the refuge of a processing product and prevents loading, and its effective manufacture approach, being able to prevent generating of a crack as much as possible, and making a spontaneous generation operation of an abrasive grain discover actively.

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EFFECT OF THE INVENTION

[Effect of the Invention] In the superfines silica grinding stone concerning this invention, mirror grinding can be carried out with high precision by fabricating a superfines silica to 50 - 80% especially 0 - 80% of void contents by using thermosetting phenol resin as a binder, without carrying out loading of the abrasives-ed, such as a silicon wafer. About the mixed rate to the superfines silica of phenol resin, if it is especially made 5 - 15vol %, it will become convenient at 1-99vol % and the point which can prevent blinding by omission of a reaction silica abrasive grain. Moreover, if Vickers hardness is especially set to 1-10Hv one to 40 Hv, it is convenient at the point that grinding [****** / soft] can be performed and grinding learned from the configuration of a work material can be performed. Degree of concentration is convenient at the point which can form a chip pocket and prevents blinding 50-300, and by carrying out to 50 to about 150 especially. Moreover, it can fabricate by this invention approach to the grinding stone of a necessary configuration by carrying out press forming of the molding material with metal mold in a vacuum housing, without generating a crack and a crack. In case a Plastic solid is especially picked out from metal mold, generating of a crack can be lessened as much as possible by making it take out, applying pressurization to a Plastic solid.

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MEANS

[Means for Solving the Problem] This invention is equipped with the next configuration in order to attain the above-mentioned purpose. That is, the superfines silica grinding stone concerning this invention is characterized by having carried out press forming of the superfines silica coating was carried out [the silica] by phenol resin, and forming it in 0 – 80% of void contents. It is suitable if the rate of phenol resin to a superfines silica makes it 1 – 99vol %. The Vickers hardness number of a grinding stone is suitable in it being 1-50Hv. Moreover, degree of concentration is suitable in it being 50-300. By the manufacture approach of the superfines silica grinding stone concerning this invention The process which adds a superfines silica into the liquid which dissolved thermosetting phenol resin in the volatile solvent, makes the shape of a slurry, is stirred and is mixed to homogeneity, The process which flies a volatile component and is dried from this mixed liquor object, and a dry matter are ground. The process which forms the powder-like molding material with which coating of the superfines silica was carried out by phenol resin, It is characterized by providing the process which carries out press forming of said molding material to a necessary configuration with metal mold within the container made into the shape of a vacuum, and the process which the acquired Plastic solid is calcinated [process] and stiffens phenol resin. If it is made to take out, applying pressurization to a Plastic solid in case a Plastic solid is picked out from metal mold, a crack does not occur in a Plastic solid but it is suitable for it. It is the compacting pressure at the time of carrying out press forming of said molding material 50 – 6000 kgf/cm2 It is suitable if it sets up. It is suitable if the rate of thermosetting phenol resin to a superfines silica is made into 1 – 99vol %.

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OPERATION

[Function] Mirror grinding can be carried out with high precision by fabricating a superfines silica in the superfines silica grinding stone concerning this invention to 0 – 80% of void contents by using thermosetting phenol resin as a binder, without carrying out loading of the abrasives-ed, such as a silicon wafer. If the mixed rate to the superfines silica of phenol resin is made into 5 – 15vol %, it will become convenient in that the silica abrasive grain to which it reacted after mechanochemical polishing is omitted. Moreover, if Vickers hardness is set to 1-10Hv, when grinding [****** / soft] can be performed and the work material has curved, it is convenient at the point that grinding learned from the configuration can be performed. Degree of concentration is convenient by carrying out to 50 to about 150 at the point which can form a chip pocket and prevents blinding. Moreover, it can fabricate by this invention approach to the grinding stone of a necessary configuration by carrying out press forming of the molding material with metal mold in a vacuum housing, without generating a crack and a crack. In case a Plastic solid is especially picked out from metal mold, generating of a crack can be lessened as much as possible by making it take out, applying pressurization to a Plastic solid.

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EXAMPLE

[Example] Hereafter, the suitable example of this invention is explained to a detail based on an accompanying drawing. The superfines (ultrafine particle powder) silica used by this example is the thing of the anhydrous silica (Aerosil: product made from Japanese Aerosil, Inc.) with the smallest particle diameter in the silica (SiO2) which is generated by a gaseous-phase method, especially flame hydrolysis, and is manufactured based on current British Standard of a high grade. Table 1 shows the class and specification of the used abrasive grain. It turns out that it is a very detailed particle with a particle size [of the particle size of 40nm (No.OX50), 16nm (No.130), and 7nm (No.380)] of 40nm or less. These particles form the network structure which the almost globular form primary particle had lightly, and do not exist in the state of an independent particle in fact. However, in the case of 40nm particle size, it is an exception, and it can be seen clearly. [of the independent globular form primary particle]
[0008] On the other hand, the binder selected thermosetting phenol resin by the following reasons.
** In the middle of processing, melt into water (grinding fluid) and the configuration of a grinding stone does not collapse.
** Heat can be applied and hardened after mixing and fabricating an abrasive grain and a binder. Table 2 shows the specification of the PR-50696 thermosetting phenol resin (product made from Sumitomo DEYURESU, Inc.) which is the used binder. A volatile component is the thing of the liquid condition gelled after 63-second progress by 51.7vol(s) % and 165 **.
[0009] In the cause whose manufacture of a superfines silica grinding stone was not completed, uneven mixing with the binder by condensation, conglomerate, etc. of a particle must be solved. Then, it noted mixing in the state of the liquid which gave dispersibility to the condensed particle as much as possible. That is, as shown in drawing 1 , after melting thermosetting phenol resin [18g each (13.8vol %), 40g (26.2vol %), 80g (41.5vol %), and 124g (52.4vol%), 207g (64.7vol%)] by a lot of volatile solvents (for example, ethyl alcohol) (1200 g), 100g of superfines silica powder was added, and it was made the shape of a slurry. It mixed so that it might stir for about 10 minutes with a screw after that and a binder and a silica particle might become homogeneity. It took care enough that a foreign matter etc. did not mix at that time.
[0010] Then, in order to fly and lose a volatile component from the mixed liquor object condition of a superfines silica, phenol resin, and ethyl alcohol, 50 degrees C was dried with the vacuum dryer for about 2 hours. Completely unlike the silica powder before mixing carried out lightly, the dried mixed powder has become hard lightly in the condition of having carried out rustlingly. This was ground in the mortar and it was made the shape of powder. Phenol resin is in the condition that coating was mostly carried out to homogeneity around the silica particle. Thus, the molding material has been adjusted.
[0011] Drawing 2 shows the outline of shaping equipment 10. 12 is a body container, and if it is formed with transparent acrylic resin etc. so that an internal state may be in sight from the outside, it is suitable. A body container 12 inferior-surface-of-tongue side is sealed by the bottom plate 14 free [removal]. 16 is an O ring. Moreover, 18 is pressed with the rod 20 of press equipment, and is a movable movable head about the inside of the body container 12. A movable head 18 may be fixed to rod 20 lower limit. 19 is an O ring. 21 is metal mold which

consists of cemented carbide, and has a dice 22, bottom punch 28, and upper punch 30. The through tube 24 penetrated in the vertical direction is formed in a dice 22, on the spring 26 arranged on a bottom plate 14, balance is maintained and installation has become possible. Bottom punch 28 is arranged on a bottom plate 14, and the upper part is carrying out ** ON to the through tube 24 from the lower part. The lower part inserts upper punch 30 in a through tube 24 from the upper part. Upper punch 30 may be fixed to movable head 18 inferior surface of tongue. A cavity 32 is formed between a bottom punch 28 upper-limit side and an upper punch 30 lower-limit side.

[0012] The suction hole 34 which opens the body container 12 and the exterior for free passage is formed in the bottom plate 14, the vacuum pump which is not illustrated through a hose is connected to this suction hole 34, and the inside of the body container 12 is made to a vacua by eliminating the air in the body container 12. Upper punch 30 is formed in the dimension longer than the height of a dice 22 in order to project mold goods from a dice 22. Drawing 3 shows pressurization equipment 36. the pressurization arranged between two or more press rods 38 which contact the dice 22 top-face side of the metal mold 21 laid on the proper base as this pressurization equipment 36 was picked out from the body container 12 after shaping and upper punch 30 came to the bottom, the press plate 40 which connects a press rod 38 upper-limit side, and press plate 40 inferior surface of tongue and bottom punch 28 — it has a spring 42. 44 is a shock absorber.

[0013] A grinding stone can be fabricated as follows using the above-mentioned shaping equipment 10. In shaping equipment 10, a movable head 18 and upper punch 30 are first removed from shaping equipment. It is specified quantity ON **** in the cavity 32 wide opened in the molding material which the binder and the superfines silica were mixed as mentioned above, and was adjusted in the shape of powder. Next, after inserting upper punch 30 in a through tube 24, a movable head 18 is moved to the location near the top face of upper punch 30 as much as possible, and the body container 12 is sealed. It lengthens with a vacuum pump after that, and the inside of the body container 12 is made into a vacua, and subsequently a molding material is pressurized through a movable head 18 and upper punch 30 with press equipment, and it fabricates. The air in a molding material is fabricated without involving in air into a molding material, since it is drawn in by the vacuum pump.

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** Although there is variation a little compared with the powder before mixing, on the whole, as for mixed silica powder, a consistency and a void content show a low inclination. For this, the true specific gravity of the phenol resin used as a binder is 1 g/cm3. With extent, it is thought that it is because it is comparatively low.
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[0017] next, drawing 6 — the particle size of 40nm — overly detailed — silica powder No.OX50 The void content property over the compacting pressure at the time of coating phenol resin with each rate is shown. As compared with what does not coat a binder, the hole has decreased according to the increment in compacting pressure, and the increment in the amount of binders. Distribution of a void content is crossed to the very large range 10-70vol %. The degree of concentration and the grain ratio of an abrasive grain are converted and shown in drawing 7 from a void content. Although it is possible for what has more binders than the void content and degree-of-concentration property of a grinding stone to lessen a hole according to compacting pressure, the increment rates of among the recentralization whenever are few things. In amount of binders 13.8vol %, compacting pressure is 1000 kgf/cm3. In the field to cross, the degree of concentration of an abrasive grain has exceeded slightly from what does not coat a binder.
[0018] The result of having measured the hardness of a grinding stone by 200g of loads with the micro Vickers hardness meter to drawing 8 is shown. According to compacting pressure, the hardness of a grinding stone, i.e., degree of coupling, was freely controllable between Hv(s) 5-28. Hardness is decreasing conversely in the range in which the amount of binders exceeds [compacting pressure] 30vol(s) % by the case of 500 kgf/cm2 of being low. As shown in these, a Plastic solid (grinding stone) has the description that the grinding stone of desired degree of concentration, a void content, and degree of coupling can be manufactured, by adjusting the amount of binders, and compacting pressure. incidentally — silica particle (OX50) with a particle size of 40nm a case — a binder — coating — **** — if — the inside of a vacuum — it is not — even if — usual — the grinding stone has been fabricated by the press-forming method.
[0019] Next, the superfines silica grinding stone was manufactured and the process condition and the grinding property of a grinding stone were investigated. An abrasive grain is comparatively coarse No.OX50 called the mean particle diameter of 40nm. After adding phenol resin and mixing, in order to create the thing of grinding stone outer-diameter 150 mm and a cup mold with a width of face of 10mm and to raise the configuration precision of a grinding stone before processing using what was fabricated and calcinated, truing was performed by the diamond wheel of grain-size #1200. The method near the constant-pressure cut grinding which does not have a limit in particle size mostly as infield grinding probably performed application to the mirror grinding of a superfines silica grinding stone. After attaching **-ed material in the surface plate of a single side lapping machine (a RAPOTE star MG773B mold, Maruto Make) and attaching a grinding stone in a work-piece shaft so that the mirror plane condition of the silicon

wafer which is a workpiece can be checked also in processing, both made it rotate. Under the present circumstances, a grinding stone carries out oscillation movement to right and left, always putting a fixed pressure on **-ed material. By the diamond wheel of grain-size #1200, **-ed material was on board in the diameter student silicon wafer of 3-6 inches, and performed mirror plane processing after Taira figuring.

[0020] As a mirror grinding property of a silicon wafer, about field coarseness, it is a non-contact type surface roughness meter (WYKO:TOPO-2D, product made from WYKO), and is the evaluation die length 324. It measured by mum and wavelength 647.7 nm, and a three-point indicating micrometer estimated the grinding ratio. In the mirror grinding by the superfines grinding stone, there are few chip pockets and it is easy to produce loading. Then, in order to make it such blinding not happen, the number of chip pockets was made to increase and the thing of a porosity condition was applied as much as possible. The machined surface granularity and the grinding ratio which were obtained by the grinding stone shown in drawing 9 in the range of 45 which is a comparatively high void content - 55vol % are shown. Although machined surface granularity improved so that the void content became high, above void content 50vol %, there is no big change and about Ra1nm was maintained. Moreover, although the grinding ratio approached 1 up to void content 50vol % order, when it was made high more than it, it decreased gradually. This is making a void content to some extent high, and is considered to be the condition that removal of the resultant by processing became easy, when it is the almost same degree of coupling, removal efficiency also has the high one where the degree of concentration of an abrasive grain is higher, and it is thought that the effectiveness of also making surface roughness improving arises.

[0021] Drawing 10 shows the effect the degree of coupling of the superfines silica grinding stone of this example affects a mirror grinding property. In the volume rate 7 of a binder - 19vol % range which are expected to have a comparatively high void content and abrasive grain degree of concentration, surface roughness and a grinding ratio became high, so that degree of coupling became low. Although the silica grinding stone of degree of coupling higher than this range was applied, loading arose firmly and processing of long duration was difficult. Therefore, it can be said that the superfines silica grinding stone of this example becomes what has the one of a high void content, quantity degree of concentration, and low degree of coupling more suitable [the mirror grinding property of a silicon wafer].

[0022]

[Table 1]

NO.	O×50	130	380
表面積 (m^2/g)	50±15	120±20	380±30
粒子サイズ (nm)	40	16	7
見掛け比重 (g/l)	80	60	60
真比重 (g/cm^3)	2.2		

[0023]

[Table 2]

比重 (g/l)	1.026
粘度 (poise)	0.2
ゲル化温度時間	165℃, 63sec
不揮発性成分(vol%)	48.3

[0024]

[Table 3]

ﾊﾞｲﾝﾀﾞｰ 量 (vol%)	ｼﾘｶ 微粉末の重量 (g)	ﾊﾞｲﾝﾀﾞｰ 重量 (g)	混合物の真重量 (g/cm^3)
0	—	—	2.20
13.8	50	9	2.06
26.2	50	20	1.94
41.5	50	40	1.79

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1.This document has been translated by computer. So the translation may not reflect the original precisely.
2.**** shows the word which can not be translated.
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DESCRIPTION OF DRAWINGS

[Brief Description of the Drawings]
[Drawing 1] It is manufacture process drawing of a grinding stone.
[Drawing 2] It is the sectional view showing the outline of shaping equipment.
[Drawing 3] It is the explanatory view showing the outline of pressurization equipment.
[Drawing 4] It is the graph which shows the consistency of the Plastic solid at the time of carrying out pressing by 500 kgf/cm2.
[Drawing 5] It is the graph which shows the void content of the Plastic solid at the time of carrying out pressing by 500 kgf/cm2.
[Drawing 6] It is the graph which shows the void content property over compacting pressure.
[Drawing 7] It is the graph which shows the degree of concentration and the grain ratio of an abrasive grain.
[Drawing 8] It is the graph which shows the degree of coupling (hardness) of a grinding stone.
[Drawing 9] It is the graph which shows machined surface granularity and a grinding ratio.
[Drawing 10] The degree of coupling of a grinding stone is the graph which shows the effect affect a mirror grinding property.
[Description of Notations]
10 Shaping Equipment
12 Body Container
14 Bottom Plate
18 Movable Head
21 Metal Mold
22 Dice
24 Through Tube
26 Spring
28 Bottom Punch
30 Upper Punch
32 Cavity
36 Pressurization Equipment
38 Press Rod
40 Press Plate
42 Pressurization — Spring
46 Plastic Solid

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